

# PATENT SPECIFICATION

(11) 1 333 475

1 333 475

## NO DRAWINGS

- (21) Application No. 13493/71 (22) Filed 6 May 1971  
 (31) Convention Application No. P 20 24 051.1  
 (32) Filed 16 May 1970 in  
 (33) Germany (DT)  
 (44) Complete Specification published 10 Oct. 1973  
 (51) International Classification A61K 7/00; C11D 1/74  
 (52) Index at acceptance  
 A5B 771 774  
 C5D 6B11A 6B11C 6B12B1 6B12F1 6B12F2 6B12L  
 6B12N4 6B12NX 6B4 6C8



## (54) COSMETIC COMPOSITIONS

(71) We, HENKEL & CIE, GMBH, a German Company, of 67, Henkelstrasse, Duesseldorf, 4000, Germany, do hereby declare the invention, for which we pray that a patent may be granted to us, and the method by which it is to be performed, to be particularly described in and by the following statement:—

The invention relates to a composition for use in cosmetic preparations, especially cosmetic cleansing agents, containing substances for replacing the oil in the skin, based on esterification products of glycerine-ethylene oxide adducts with long-chain fatty acids.

Cosmetic cleansing agents such as shampoos, foam baths, toilet soaps and similar products cause a more or less great removal of oil from the skin on repeated use. This phenomenon is particularly pronounced when the cleansing agents are based on synthetic, surface-active substances such as alkylbenzene-sulphonates, fatty alcohol sulphates, olefine sulphonates, fatty alcohol ether sulphates and other surface-active compounds. Attempts have therefore been made to neutralise this removal of oil from the skin by replacing the oil by means of suitable additions to the cleansing agents. Allowance has in such case to be made for disadvantages, however, since the products generally concerned in replacing the oil in the skin have an unfavourable action on the foaming properties of the cleansing agents and, in cosmetic preparations based on alcoholwater mixtures, show insufficient solubility.

The present invention provides a composition for use in cosmetic preparation which comprises a surface active compound and the esterification product of an ethylene oxide addition compound produced from glycerine and 4 to 20 mol of ethylene oxide per mol of glycerine, with a fatty acid of chain length

from 8 to 18 carbon atoms in a ratio of 1 to 2 mol of fatty acid to 1 mol of glycerine-ethylene oxide addition compound as oil replacement material.

Esterification products of ethylene oxide addition compounds produced from glycerine and 7 to 15 mol of ethylene oxide per mol of glycerine with fatty acids of chain length from 8 to 18 carbon atoms in a ratio of 1 mol of fatty acid to 1 mol of glycerine-ethylene oxide addition compound are preferred as the oil replacement materials.

The preparation of the ethylene oxide addition compound as intermediate product was generally effected in known way by reacting glycerine with ethylene oxide in the desired proportions with alkaline catalysis by means of sodium ethylate. For the further treatment, the ethylene oxide addition compound obtained was reacted in the usual way with a fatty acid of chain length from 8 to 18 carbon atoms in the molar ratio of 1:1 or 1:2, using isopropyl titanate as esterification catalyst. The esterification products obtained were light-coloured to yellowish liquids of low viscosity with an oil character to lard-like products of a faint self colour.

The quantities of oil replacement material according to the invention used in the cosmetic preparations may vary within very wide limits according to the product and its oil-removing action, and generally vary from 2 to 50% by weight, especially 5 to 25% by weight. Still higher additions are possible if the esterification products according to the invention are used at the same time in their property as surface-active substances, but in most cases this use will be of small advantage.

Oil replacement materials to be used according to the invention include, for example, esterification products from

				the adduct of 1 mol of glycerine + 4 mol of ethylene oxide with 1 mol of coconut fatty acid $C_{11-18}$
		"	"	+ 6 mol of ethylene oxide with 2 mol of coconut fatty acid $C_{11-18}$
5		"	"	+ 7 mol of ethylene oxide with 1 mol of coconut fatty acid $C_{11-18}$
		"	"	+ 7 mol of ethylene oxide with 1 mol of tallow fatty acid
		"	"	+ 8 mol of ethylene oxide with 1 mol of oleic acid
10		"	"	+ 9 mol of ethylene oxide with 1 mol of palm kernel fatty acid
		"	"	+ 10 mol of ethylene oxide with 1 mol of tallow fatty acid
15		"	"	+ 10 mol of ethylene oxide with 2 mol of groundnut oil fatty acid
		"	"	+ 12 mol of ethylene oxide with 1 mol of coconut fatty acid $C_{11-18}$
		"	"	+ 15 mol of ethylene oxide with 1 mol of palm kernel fatty acid
20		"	"	+ 15 mol of ethylene oxide with 2 mol of tallow fatty acid

25 The present invention will be further described by way of illustration with reference to the following examples. Abbreviations and units used in the examples are defined as follows:—

30 "Acid value" is the number of mg of potassium hydroxide which are needed to neutralize the free esterified fatty acid contained in 1 g of substance;

35 "Saponification value" is the number of mg of potassium hydroxide which are needed to completely saponify 1 g of ester;

40 "Hydroxyl number" is the number of mg of potassium hydroxide which are needed to neutralize the acetic acid which is absorbed by 1 g of material. The procedure used is as follows; the substance being investigated is weighed out exactly and is then acetylated by heating with crystalline acetic acid, all the OH groups present thereby being esterified. Subsequently, the acetylated portion of the material is separated by a shaking and washing procedure and the acetylated part is saponified using boiling potassium hydroxide solution. The excess potassium hydroxide which is present after the saponification steps is determined by titration with hydrochloric acid.

"EO" is the ethylene oxide group;

"WAS" is active washing substance.

All parts used throughout the examples are by weight unless otherwise specified.

#### Examples

The following esterification products were used for the experiments and cosmetic preparations described below.

- (A) (1 mol of glycerine + 7.4 mol of ethylene oxide) with 1 mol of coconut fatty acid  $C_{11-18}$  Acid value 1.0, sap. value 92, hydroxy value 185
- (B) (1 mol glycerine + 7.4 mol of ethylene oxide) with 1 mol of tallow fatty acid (Acid value 1.1, sap. value 83, hydroxy value 166)
- (C) (1 mol glycerine + 10 mol of ethylene oxide) with 1 mol of tallow fatty acid (Acid value 1.4, sap. value 71, hydroxy value 141)

Since for cosmetic cleansing compositions the ability to combine with certain surface-active compounds is of essential importance, mixtures given in the following Table were tested.

TABLE I

Mixing component	Mixture 1	Mixture 2	Mixture 3
(A)	10	—	—
(B)	—	10	—
(C)	—	—	10
Sodium lauryl ether sulphate (2 EO) (27—28% WAS)	50	50	50
Water	40	40	40
Result	Clear homogeneous solution	Clear homogeneous solution	Clear homogeneous solution

In a further experiment the foaming power of a foam bath basic recipe with additions of the individual oil replacement materials was examined.

TABLE II

Components	Mixture 1	Mixture 2	Mixture 3	Mixture 4	Mixture 5
Sodium lauryl ether sulphate (2 EO) (27—28% WAS)	60	60	60	60	60
Sodium lauryl sulphate (over 90% WAS)	5	5	5	5	5
Isopropyl myristate	—	5	—	—	—
(A)	—	—	5	—	—
(B)	—	—	—	5	—
(C)	—	—	—	—	5
Water	35	30	30	30	30
Appearance	clear	turbid deposit formed	clear	clear	clear
<b>Foaming power</b>					
Initial volume in ml 1 minute after beating					
0.5 g/litre	260	170	300	340	240
1.0 g/litre	510	250	490	490	400
2.0 g/litre	660	330	610	640	540
Breaking down of the volume of foam in ml/minute					
0.5 g/litre	2.0	2.5	4.0	4.5	1.5
1.0 g/litre	5.5	3.5	7.0	8.0	4.0
2.0 g/litre	8.0	6.5	9.5	3.0	6.5

5 The foaming power of the individual mix-  
tures was measured in the foam-beating  
machine according to DIN (Deutsche Indus-  
trie Norm) classification No. 53,902, in which  
the volume of foam was measured. The figures  
for the foam were taken at 45°C in water of  
10° German hardness after 30 beats. The  
measurement was taken 1 minute after the  
end of the beating and 21 minutes after the  
end of the beating. The breakdown of the  
10 volume of foam was calculated in ml/minute  
from the decrease of the volume of foam in

20 minutes. The amounts given in g/litre  
relate to the respective mixture.

As may be seen from the above Table, the  
foam values of the mixtures containing oil  
replacement means according to the invention  
are substantially better than when isopropyl  
myristate is used as oil replacement materials,  
and are scarcely inferior to the figures for a  
mixture of pure detergent substances.

A few formulations for cosmetic preparations  
containing oil replacement materials according  
to the invention are given below.

15

20

25

Clear Shampoo

Sodium lauryl sulphate (2EO) (27—28% WAS)	40	parts by weight		
Coconut fatty acid diethanolamide	6	"	"	"
Oil replacement means (A)	10	"	"	"
Water	44	"	"	"

Shampoo for dry hair

Sodium lauryl ether sulphate (2 EO) (27—28% WAS)	20	parts by weight		
Sodium lauryl sulphate (90% WAS)	5	"	"	"
Coconut fatty acid diethanolamide	3	"	"	"
Coconut fatty acid monoethanol- amide paste 30%	5	"	"	"
Water-soluble vitamin F	0.5	"	"	"
Oil replacement means (B)	25.0	"	"	"
Water	41.5	"	"	"

Foam bath

Sodium lauryl ether sulphate (2 EO) (27—28% WAS)	30	parts by weight		
Sodium lauryl sulphate (90% WAS)	15	"	"	"
Coconut fatty acid diethanolamide	5	"	"	"
Pine-needle oil	5	"	"	"
Oil replacement means (C)	10	"	"	"
Water	35	"	"	"

Hair wash

Isopropanol	60.0	parts by weight		
Menthol	0.2	"	"	"
Calcium pantothenate	0.05	"	"	"
Vitamin H	0.30	"	"	"
Inositol	0.10	"	"	"
Perfume	0.50	"	"	"
Oil replacement means (C)	5.00	"	"	"
Water	33.85	"	"	"

After-shave lotion

Ethyl alcohol 96%	65.0	parts by weight		
Menthol	0.2	"	"	"
Camphor	0.2	"	"	"
Peruvian balsam	0.1	"	"	"
Perfume	0.5	"	"	"
Glycerine	5.0	"	"	"
Witch hazel extract	10.0	"	"	"
Boric acid	0.5	"	"	"
Oil replacement means (A)	10.0	"	"	"
Water	8.5	"	"	"

## Sun tan cream

Colloidally dispersed mixture of 90  
parts of cetylstearyl alcohol  
and 10 parts of sodium lauryl  
sulphate

2-Octyldodecanol

Groundnut oil

Light-protective means

Oil replacement means (B)

Water

10.0 parts by weight

10 " " "

5 " " "

2 " " "

20 " " "

53 " " "

The oil replacement compositions according to the invention can be used particularly advantageously in cosmetic cleansing means, because they do not exert any appreciable influence on the foaming power of the surface-active products, and because they already have a good solubility in alcohol-water mixtures.

# WHAT WE CLAIM IS:—

1. A composition, for use in cosmetic preparations, which comprises a surface active compound and the esterification product of an ethylene oxide addition compound produced from glycerine and 4 to 20 mol of ethylene oxide per mol of glycerine with a fatty acid of chain length from 8 to 18 carbon atoms in a ratio of 1 to 2 mol of fatty acid to 1 mol of glycerine-ethylene oxide addition compound as oil replacement material.

2. A composition according to claim 1, in which the ratio of glycerine to ethylene oxide in the addition compound is from 7 to 15 mols

of ethylene oxide per mol of glycerine.

3. A composition according to claim 1 or 2 wherein the fatty acids of chain length from 8 to 18 carbon atoms are in a ratio of 1 mol of fatty acid to 1 mol of the glycerine-ethylene oxide addition compound.

4. A composition according to claims 1 to 3, containing from 2 to 50% by weight of the oil replacement material.

5. A composition according to claim 1 to 4 containing from 5 to 25% by weight of the oil replacement material.

6. A composition according to claim 1 substantially as hereinbefore described with reference to and as illustrated in the foregoing examples.

7. A cosmetic preparation whenever containing a composition as claimed in any one of claims 1 to 6.

W. P. THOMPSON & CO.,

12, Church Street, Liverpool, L1 3AB.

Chartered Patent Agents.